OBSERVATION OF SHEAR WAVE GENERATION AT BONDED SILICON-CARBIDE INTERFACE

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INTRODUCTION

The drive toward higher efficiencies and performance in energy production and transportation equipment has posed a continual challenge for the materials scientists and engineers involved in design and construction of such equipment. The laws of thermodynamics specify that increased efficiency can best be achieved by the use of the highest possible temperatures. As the desired operating temperature of a system increases, the limitations of the materials being used for construction of the system place an upper limit of the temperatures and thus the efficiencies that can be achieved.

Ceramic materials show a number of potential advantages for such use. They exhibit superior high temperature capabilities as well as strength and creep resistance that make their use attractive when compared to more traditional metallic materials. Overall, the mechanical and chemical properties of ceramic materials make them ideal candidate materials for industrial uses where increased energy conversion efficiency is desirable.

The difficulty of fabrication of complex parts from ceramic materials has been a drawback to the expansion of their use. Ceramic components have been manufactured by pressing of the powders in the green state to a near net shape, sintering to obtain the final properties and machining or grinding to achieve the final dimensions. This sequence of steps somewhat limits the complexity of the component shape that can be produced. In addition, ceramic materials are inherently brittle. The presence of any undesirable flaw in the final part will have an extremely deleterious effect on the mechanical properties. Inspection for the presence of these flaws, while not an absolute necessity, is advantageous and creates additional cost and time constraints.

In this work, techniques are being developed for joining individual SiC components by the use of organic SiC precursors to create a bond between the individual components prior to final sintering. This type of joining would allow the production of much more complex shapes than is currently possible. Ultrasonic NDE techniques are being used for inspection of the joined structure for a prediction of the strength and to detect any flaws that may be present after joining. The second objective is the major thrust of this paper.

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BACKGROUND

The ceramic joints being investigated are formed by joining of two SiC components into a single final structure through the use of organic SiC precursors, in this case polycarbosilane (PCS) to form a bond prior to the final sintering of the structure. The process as currently used is: (1) Produce green state pellets from mixture of SiC and PCS, (2) Cold isostatic press (CIP) of pellets, (3) Apply joining material to pellet faces, (4) CIP of joined pellets, (5) Pyrolize joined pellets to remove binder and (6) Fire pyrolized sample for final sintering [1]. Work is still underway on the development of a final process to produce joined structures with optimum properties. Ultrasonic inspection is carried out on the fired structure to evaluate the joint nondestructively prior to destructive testing using a 4-point bend test.

The ultrasonic inspection uses a normal incidence, focused ultrasonic beam in a pulse-echo mode to obtain reflections from the joined interface. The transducer is pulsed with a spike pulse producing a broad-band ultrasonic pulse in order to facilitate investigation of the frequency response in the joint reflections. It has been shown in previous work on diffusion bonds that the amplitude of the reflected pulse provides an indication of the strength of the bond [2-3] with the total energy of the reflection providing more sensitivity [4].

Use of this technique on the joints prepared here yielded similar results on the initial set of samples examined. As was reported previously, a set of samples examined both nondestructively and destructively showed a promising trend in the data as shown in Table I. For those samples examined nondestructively, a second reflection was observed emanating from the joint area. This echo was postulated to occur due to mode conversion at the joint line. Both echoes showed sensitivity to the obtained strength with the ratio of the echo energies yielding values that appeared to be strongly correlated with the strength.

The predominant flaw type in this initial set of samples was a "shrinkage-type" defect exhibiting a crack-like shape oriented perpendicular to the joint line. This type and orientation of flaw is particularly unfavorable for inspection using a longitudinal wave since the flaw presents its smallest dimension to the wave. The second echo observed, if truly from a mode conversion at the joint line, would potentially have greater sensitivity to the flaw since the atom motion after conversion to a transverse wave would be in the direction of the flaw's greatest dimension.

SOURCE OF SECOND REFLECTED ECHO

In order to definitely determine the source for the second echo being observed reflected from the joint line, acoustic velocity measurements were made on a solid SiC

Table I. Preliminary correlation of SiC joint samples.

Sample	Density g/cm^3	UT Reflection Energy #1	UT Reflection Energy #2	UT Energy Ratio	Strength MPa
M-11-44-4		1.01×10^{-2}	3.71×10^{-3}	2.72	Very Weak
M-11-65-3		2.23×10^{-3}	2.25×10^{-4}	9.89	130
JZ-12-2	2.663	3.54×10^{-4}	3.16×10^{-7}	1118	233
Control (no joint)					196
Commercial	3.095				300-400

specimen using both longitudinal and shear wave transducers. The velocities obtained were 10,300 m/sec for longitudinal waves and 6,510 m/sec for shear waves. Using these velocities and the known geometries of the joined SiC samples, simple timing predictions of the round-trip transit time to the joint interface were made and compared with the observed echoes from the SiC samples. These results and comparisons are shown in Table II. The relatively good agreement seen in Table II indicates that the observed echoes are indeed from; #1 - longitudinal wave propagating in both the incident and reflected leg, and #2 - longitudinal wave propagating in the incident leg and transverse wave propagating in the reflected leg with the transverse wave being created by mode conversion at the joint interface.

Observation of similar echoes have been made in other transducer-material combinations and are believed to be a result of the focusing of the ultrasonic wave resulting in off-normal portions of the wave being incident on the reflecting surface. A number of other such wave trains containing this longitudinal-transverse (L-T) echo are shown in Figure 1 for combinations of focused transducers in SiC and steel. For each wavetrain, the front surface echo and two reflected echoes are shown. The L-T echo is indicated in each case by an arrow on the wavetrain. The L-T echo can be seen in all four examples shown, however the relative amplitudes of the two reflected echoes (L-L and L-T) are widely different for all cases except the 1.27 cm, Focus = 10 cm example where the L-L and L-T echoes are similar in amplitude within a factor of 2-3 whereas the two echoes in the other examples shown vary by a factor of 7 or more with the other closest example also occurring in SiC.

The generation of a mode-converted wave depends on the presence of non-normal incidence waves for the conversion to a transverse wave to occur from the longitudinal wave that is incident on the reflection surface. It is apparent that in the case of SiC, much more of the incident longitudinal wave is converted to a transverse wave than in the case of steel. This can be explained using a comparison of the ultrasonic velocities and ray tracing to determine the angle of the included cone of ultrasonic rays incident on the surface responsible for the conversion. A simple ray tracing diagram as shown in Figure 2 illustrates the angle of the included cone for the case of a transducer focused on the back surface of a specimen. The angle of this included cone, α, is dependent on the focal length of the transducer, ultrasonic velocity of the material and thickness of the material. The included cone angle for three of the examples shown in Figure 1 are given in Table III.

As can be seen from the large included angle (63.85°) for the 10 cm focal transducer incident on the back surface of the SiC sample being investigated here, the observation of

Table II. Comparison of reflected echo times.

Sample Number	Predicted Time μsec	Observed Time μsec
1	2.47	2.57
2	3.18	3.36

Table III. Included cone angles for material-transducer combination.

Material	Thickness cm	Ultrasonic Velocity m/sec	Focal Length cm	Included Angle degrees
SiC	1.27	10,300	10.16	63.85
SiC	1.27	10,300	20.32	32.30
Steel	0.953	5,960	10.16	30.22

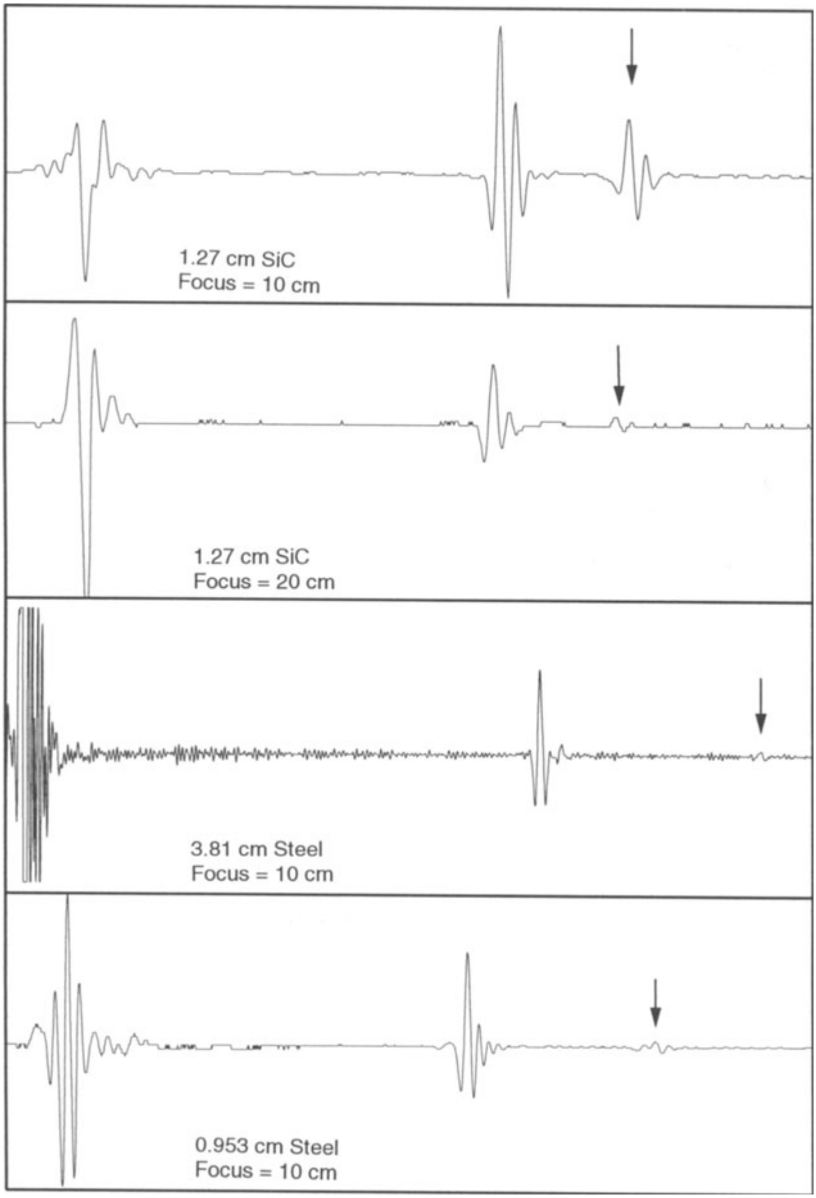

Figure 1. Wave train examples illustrating presence of L-T echo from reflecting surface

significant mode converted energy is considerably more likely than for the other combinations investigated. As was noted in the earlier work, this mode converted transverse wave is much more favorably oriented for detection and characterization of the type of defect structure seen in those preliminary joints.

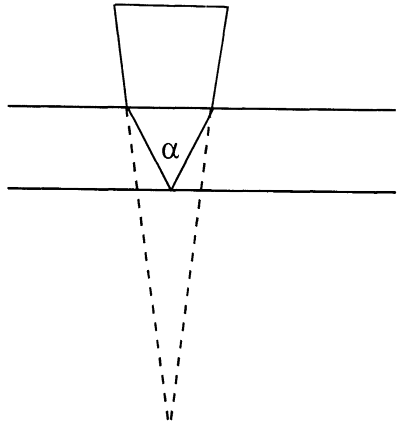

Figure 2. Ray tracing diagram defining included cone angle.

ADDITIONAL SAMPLE INVESTIGATION

A series of additional samples have been produced during investigations on optimizing the joint production process. This optimization is proceeding on the basis of both joint strength and complexity and cost of the production process. To date, the optimum production process consists of:

Blending SiC powder with 5 vol% PCS
Dry slurry and sieve through 100 mesh
Press into pellets with 44 MPa uniaxial pressure
Join pellets with 1:1 mixture by volume SiC and PCS
CIP joined pellets (5 minutes at 175 MPa)
Sinter 1 hour at 2100° C under flowing Argon

At this point, it appears that the CIPing step is the most important step in the process with the pressure playing an important part in the quality of the joint. To verify this, a series of samples were produced with the single difference being the pressure during the CIP step. These four samples were produced at pressures of 43.75, 87.5, 131.25 and 175 MPa respectively to quantify the effect of changes in pressure both on the nondestructive evaluation and the strength as determined by destructive tests.

Results from the nondestructive investigation of these samples are shown in Figure 3. These results are given in the form of contour maps of the amplitude of the reflected echo as a function of position in the sample. In general, the maps show an increasing trend of the reflection amplitude as the CIP pressure decreases with the lighter portions indicating increased reflection amplitude. All of the maps are shown with identical contour levels to facilitate comparison between them. Many of the maps exhibit a light colored ring or portion of a ring at the outer edge. This ring is due to the interaction of the ultrasonic beam with the edge of the sample and should be disregarded with the reader's attention being directed to the center portion of the map in each case. Two maps are shown for each sample with the left map being the longitudinal-longitudinal echo and the right side being the longitudinal-transverse reflection. Similar maps were drawn of the ratio of the L-L and L-T reflections but are not included since unlike the previous samples, they yielded no additional information. This lack of additional information from these ratios will be defined later.

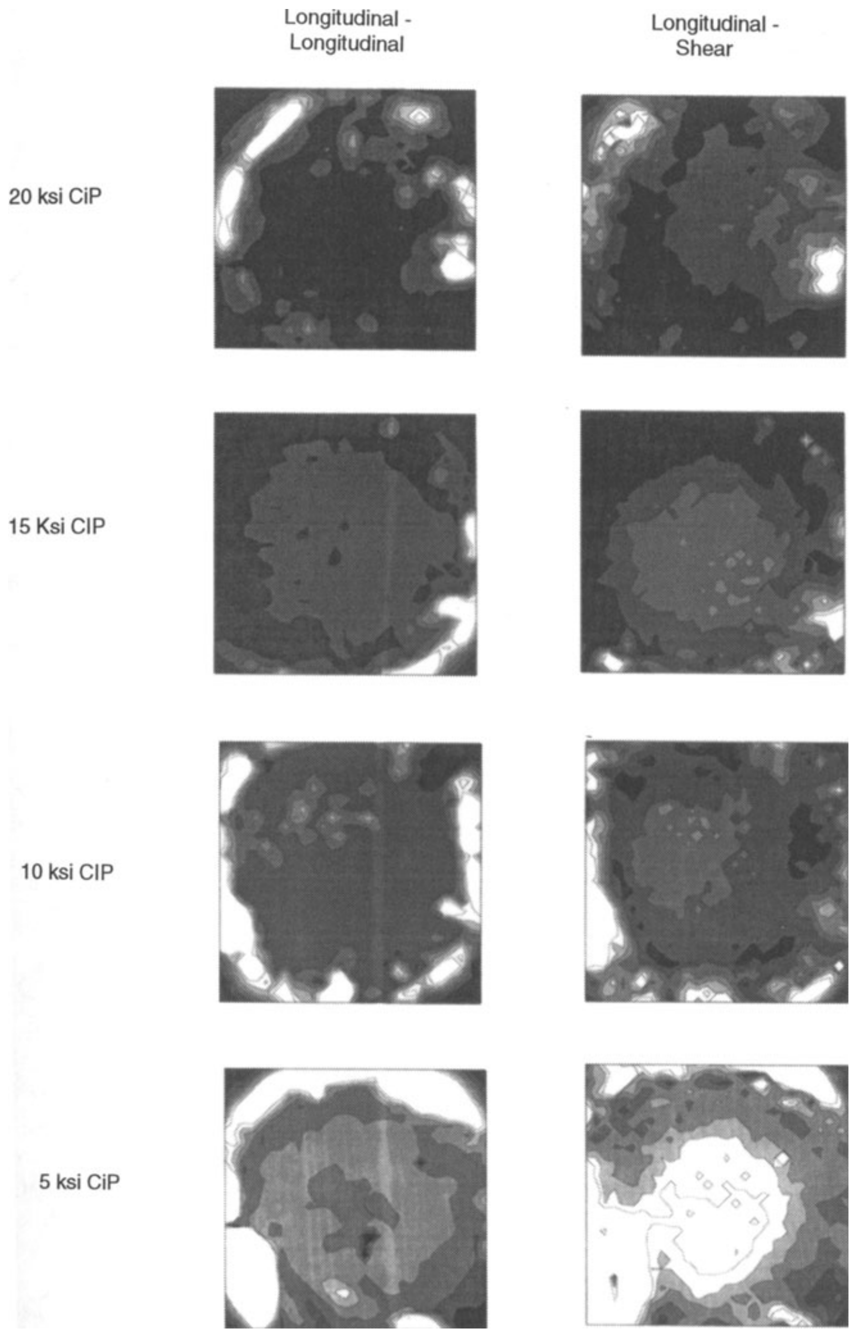

Figure 3. Ultrasonic amplitude contour maps of joints produced at varying CIP pressure.

Destructive tests were also performed on these samples with the results given in Table IV. With the exception of Sample #1 which shows an anomalously high strength, a monotonically increasing trend in the strength with the CIP pressure is seen in these samples. In general, this increasing trend in the strength can be correlated with a

Table IV. Destructive test results on variable CIP pressure samples.

Sample	CIP Pressure MPa	Strength MPa
1	43.75	267 ± 45
2	87.50	205 ± 27
3	131.25	221 ± 39
4	175.00	265 ± 15

decreasing trend in the reflection amplitudes seen in Figure 3. Examination of the samples after destructive tests showed that the predominant flaw type has changed from a "shrinkage" crack-like flaw to porosity at the joint line. This change in flaw type will explain the lack of increased sensitivity of the L-L/L-T echo ratio since a pore-type flaw does not exhibit any particular orientation and thus should not be sensitive to the direction of the atom movement during propagation of the ultrasonic beam. The strengths obtained do compare much more favorably with that found in a commercial sample as shown in Table I indicating considerable progress in development of a process for production of an optimum joint.

Additional tests for determination of initial fracture toughness measurements were also performed on the 175 MPa CIP pressure sample for comparison with a control sample with no joint. These results showed a fracture toughness for the joined sample of approximately 75% of that from the control sample with no joint. No correlation is possible with the nondestructive investigation at this time.

SUMMARY

A new sample set has been produced with strengths that begin to compare with commercial solid material. Ultrasonic monitoring of the joints being produced indicates that nondestructive evaluation is capable of a qualitative prediction of the strength of the joints being produced. Changes in the joint production process exclude quantitative nondestructive results at this time but development of a quantitative measurement appears promising. Ultrasonic monitoring of the samples being produced will continue with the intent of continuing nondestructive technique development as the joint production process proceeds toward optimum strength capability.

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REFERENCES

1. M. Akinc and J. Zheng, private communication.
2. D. D. Palmer, D. K. Rehbein, J. F. Smith and O. Buck, J. Nondes. Eval. 7, 153 (1989).
3. D. D. Palmer, D. K. Rehbein, J. F. Smith and O. Buck, J. Nondes. Eval. 7, 167 (1989).
4. G. C. Ojard, O. Buck, D. K. Rehbein and M. S. Hughes, Review of Progress in Quantitative NDE 12, edited by D. O. Thompson and D. E. Chimenti, Plenum Press, New York and London, 1993, p. 1453.